

PATENT ABSTRACTS OF JAPAN

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(54) LOW POLLUTION TYPE EMULSION FOR SINGLE LAYER ELASTIC COATING

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain an emulsion low in rain drop pollutivity and large in initial water resistance.

SOLUTION: 95-30 pts.wt. of a 1-12C acryl group-containing (meth)acrylic acid alkyl ester, 5-70 pts.wt. of styrene, 0.5-10.0 pts.wt. of an unsaturated carboxylic acid, and 0.5-10.0 pts.wt. of an amide group-containing acrylic monomer and emulsion-copolymerized in the presence of a polymerizable anionic surfactant and a polymerizable nonionic surfactant in a total amount of 0.5-10.0wt.% based on the whole monomer amount to obtain the low pollution type emulsion used for single layer elastic coating materials and having a glass transition point of -20 to 30°C.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] About the synthetic-resin emulsion from which this invention serves as a binder of a monolayer elastic coating, the raindrops-proof stain-resistance of the monolayer monolayer elastic paint film using this emulsion is good, and it is excellent also in the water resisting property in early stages of desiccation.

[0002]

[Description of the Prior Art] Since crack flattery nature and high gloss are provided by finishing of a monolayer, a lysine etc. repaints and comes out and many monolayer elastic coatings are used. However, for crack flattery nature, since the paint film has a tuck softly, it has a fault of dirt or a cone. Therefore, by hardening a paint film at the sacrifice of crack flattery nature, a tuck is pressed down and the attempt of the reduction in contamination is performed. If a paint film is hardened, contamination of the whole paint film will progress in the direction reduced to some extent. However, by this approach, "raindrops contamination" generated in the part down which storm sewage tends to flow cannot be reduced. In the part which raindrops attach, and the part which is not attached, in order that the difference of dirt may clarify dramatically, this "raindrops contamination" spoils the fine sight of a building, and poses a problem. In order to reduction-ize raindrops contamination, various attempts are performed, and they are Techno-Cosmos1992 and Sep. In Vol.2 (P. 80-84), the macromolecule 44-volume May, 1995 issue (p-307), the building finish technical October, 1993 issue (P74-77), and the building finish technical January, 1995 issue (P49-52), examination which carries out hydrophilization of the paint film front face, or blends an aquosity polymer is performed. however, the water resisting property which a monolayer elastic coating should possess if the hydrophilic property of a paint film becomes high too much -- especially -- the water resisting property in early stages of desiccation -- falling -- that a paint film flows out by the rainfall after construction **** -- blistering ***** -- it may be unacquainted and a problem occurs.

[0003]

[Problem(s) to be Solved by the Invention] This invention offers the emulsion for monolayer elastic coatings which it is hard to carry out raindrops contamination, and a paint film does not flow out by the rainfall after construction, but has sufficient initial water resisting property without blistering. It is offering the emulsion for coatings which is compatible in the hydrophilic property of a paint film, and the water resisting property in early stages of desiccation, and specifically has high gloss, ductility, reinforcement, and adhesion.

[0004]

[Means for Solving the Problem] By using together a polymerization nature anionic surfactant and a polymerization nature nonionic surfactant as an emulsifier for the waterproof improvement in early stages of desiccation, and specifying the amount used, such as it, using the acrylic monomer which has an amide group as grant of a hydrophilic property, this invention solved the technical problem and completed this invention. "This invention, 1. The alkyl group content (meta) acrylic-acid alkyl ester of

carbon numbers 1-12 95-30 Weight section Styrene 5-70 Weight section Unsaturated carboxylic acid 0.5 - 10.0 weight section Amide group content acrylic monomer The emulsion for low contamination mold monolayer elastic coatings of -20-30 degrees C of glass transition points which carried out the emulsion polymerization and which were obtained using both the surfactants of 0.5 - 10.0% of the weight of a polymerization nature anionic surfactant, and a polymerization nature nonionic surfactant to all the monomers that consist of the 0.5 - 10.0 weight section.

2. Emulsion for low contamination mold monolayer elastic coatings whose alkyl group content (meta) acrylic-acid alkyl ester of carbon numbers 1-12 is 1 chosen from methyl acrylate (meta), butyl acrylate (meta), and 2-ethylhexyl acrylate (meta), or 2 or more and which was indicated by the 1st term.

3. Emulsion for low contamination mold monolayer elastic coatings indicated by the 1st term or the 2nd term whose amide group content acrylic monomers are acrylamide and/or meta-acrylamide.

4. emulsion for low contamination mold monolayer elastic coatings which added and copolymerized 1 or 2 or more among the hydroxyl content partial saturation monomer, the alkoxy silyl radical content partial saturation monomer, the glycidyl group content partial saturation monomer, and the aceto acetyl group content partial saturation monomer in the comonomer indicated by any 1 term of the 1st term thru/or the 3rd term. " -- it is related.

[0005] As acrylic-acid (meta) alkyl ester containing the alkyl group of the carbon numbers 1-12 used by this invention, a methyl acrylate (meta), an ethyl acrylate (meta), acrylic-acid (meta) propyl, butyl acrylate (meta), 2-ethylhexyl acrylate (meta), acrylic-acid (meta) cyclohexyl, acrylic-acid (meta) lauryl, etc. are raised. That to which copolymerization of the acrylic ester in which carbon numbers 1-12 carry out alkyl group content (meta) was carried out is desirable, when controlling weatherability, a water resisting property, and a glass transition point and adjusting a paint film degree of hardness, and 1 of a methyl acrylate (meta), butyl acrylate (meta), and 2-ethylhexyl acrylate or 2 or more are especially desirable. The amount used is 95 - 30 weight section. If fewer than 30 weight sections, weatherability is not enough and cannot adjust a glass transition point to desired temperature, either. if [than 95 weight sections] more -- adjustment of gloss -- carrying out -- ***** -- ** The styrene used by this invention raises gloss, and it is used in order to make a glass transition point adjust. Moreover, with styrene, it is advantageous also because of the raw material cost reduction of an emulsion. The amount used is 5 - 70 weight section. If [than 70 weight sections] more [gloss is not more enough than 5 weight sections and], weatherability will fall and the ductility of a paint film will also become low.

[0006] Since ionize the unsaturated carboxylic acid used by this invention on an alkali side, it makes an electric double layer form and stabilizes an emulsion particle, it uses, and an acrylic acid, a methacrylic acid, a maleic acid, a crotonic acid, an itaconic acid, etc. are mentioned. The amount used becomes unstable [grain children fewer than the 0.5 weight section] in the 0.5 - 10.0 weight section, and if [than the 10.0 weight sections] more, a water resisting property will serve as a defect.

[0007] The amide group content acrylic monomer used by this invention is used in order to carry out hydrophilization of the paint film front face. Although this invention person copolymerized and examined the monomer of various kinds of hydrophilic radical content, the amide group content (meta) acrylic ester of what raindrops-proof stain resistance is raised and does not have an adverse effect on the water resisting property in early stages of desiccation was best. Specifically, acrylamide, methacrylamide, N-methylol (meta) acrylamide, N,N-dimethylacrylamide, N-isopropyl acrylamide, N-t-butyl acrylamide, N-n-butoxy methylacrylamide, etc. are mentioned. They are acrylamide and methacrylamide preferably. The amount used is the 0.5 - 10.0 weight section. It is the 1.5 - 6.0 weight section preferably.

[0008] A monomer copolymerizable in addition to monomers, such as this, can be used. For example, the acrylic-acid 2-hydroxyethyl used for adjustment of a hydrophilic property, Hydroxyl content (meta) acrylic ester, such as acrylic-acid 2-hydroxymethyl and acrylic-acid (meta) hydroxypropyl, The vinyltrimetoxysilane for making the interior of a particle construct a bridge and raising a hydrophilic property and alkali resistance, Vinyltriethoxysilane, a vinyl tris (beta-methoxyethoxy) silane, Vinylmethyldimethoxysilane, gamma-methacryloxypropyl trimethoxy silane, Alkoxy silyl radical content partial saturation monomers, such as gamma-methacryloxypropylmethyldimethoxysilane and gamma-

acryloxypropyltrimethoxysilane, Glycidyl group content partial saturation monomers, such as glycidyl (meta) acrylate, Functionality monomers, such as the aceto acetyl group content partial saturation monomers, such as acetoacetoxylethyl (meta) acrylate and an acetoacetic-acid allyl compound, other ethylene, vinyl acetate, a vinyl chloride, a vinylidene chloride, etc. can be used. Moreover, the divinylbenzene which has a polymerization nature unsaturated bond two or more, diallyl phthalate, triallyl isocyanurate, tetra-allyloxy ethane, terephthalic-acid diaryl, ethylene GURIKORUJI (meta) acrylate, polyethylene GURIKORUJI (meta) acrylate, polypropylene GURIKORUJI (meta) acrylate, methoxy polyethylene GURIKORUJI (meta) acrylate, methoxy polypropylene GURIKORUJI (meta) bitter taste relay **, etc. can be used.

[0009] Although monomers, such as this, are copolymerized and emulsion polymer is obtained, the glass transition point of the polymer needs to be -20-25 degrees C. The glass transition point of a polymer influences the elongation and stain resistance of a paint film greatly, and when it is higher than 25 degrees C, the elongation of a paint film lacks it few at the crack flattery nature as an elastic coating. - If lower than 20 degrees C, the tuck of a paint film will become strong, and dirt becomes easy to adsorb and is not desirable. Although this glass transition point can be surveyed also from an emulsion film, it can ask on count by the following formula. The glass transition point specified by this invention is on count, and is called for. Formula [0010]

[Equation 1]

$$\frac{1}{Tg} = \frac{w_1}{Tg_1} + \frac{w_2}{Tg_2} + \frac{w_3}{Tg_3} + \dots + \frac{w_n}{Tg_n}$$

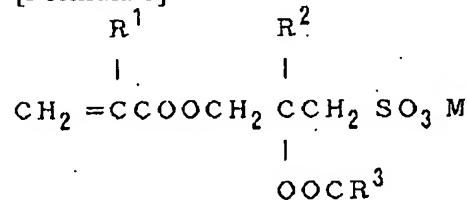
[0011]

$w_1 + w_2 + w_3 + \dots + w_n = 1$, and Tg_1, Tg_2, Tg_3 and Tg_n are the glass transition point (absolute temperature) of each homopolymer.

[0012] Next, the anionic surfactant and nonionic surfactant of polymerization nature which are used by this invention are explained. This etc. acts as an emulsifier in an emulsion polymerization, and what has a polymerization nature double bond in the structure as a polymerization nature machine is used. Since there is no free emulsifier which is not sticking to the particle front face which exists when it combines with a particle front face firmly and the usual surfactant is used by existence of this polymerization nature double bond, in order to carry out a chemical bond to other monomers, the water resisting property of a paint film is not reduced. Moreover, the description of this invention is to use anionic and nonionic together with the surfactant of polymerization nature. This is for satisfying polymerization stability and coating fitness, maintaining a high water resisting property.

[0013] Moreover, 0.5 - 10.0 % of the weight is suitable for the amount of the anionic surfactant of polymerization nature, and the nonionic surfactant used to all monomers. If fewer than 0.5 % of the weight, the emulsifier of the system of reaction will be insufficient and it will not become a good emulsion. When [than 10.0 % of the weight] more, unreacted emulsifiers increase in number and a water resisting property is made to fall. Specifically, it is [0014].

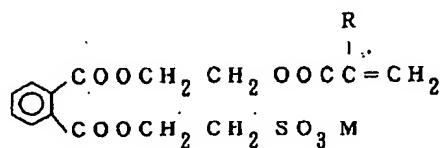
[Formula 1]



[0015] The alkyl of R1, R2:H, CH3, and R3:C 7-21, an alkenyl radical, M: Alkali metal, ammonium,

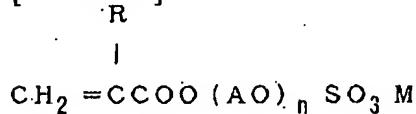
[0016]

[Formula 2]



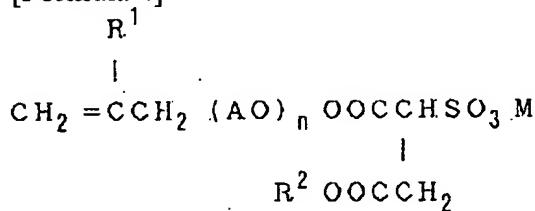
[0017] R: H, CH₃, M : alkali metal, ammonium, an amine, [0018]

[Formula 3]



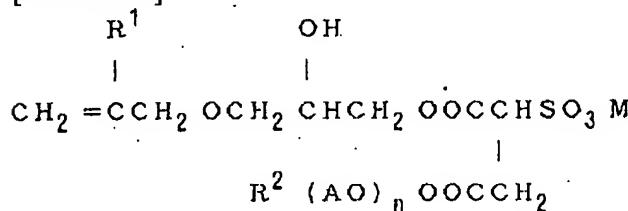
[0019] R: H, CH₃, A:alkylene group, n:2 or more integers, M : univalent, a divalent cation, [0020]

[Formula 4]



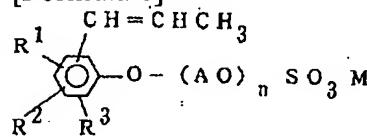
[0021] R1: H, CH₃, R2: Alkylene groups of A:C 2-4, such as unsubstituted or a permutation hydrocarbon group, a permutation alkylene group, n:0, a positive number, [0022].

[Formula 5]



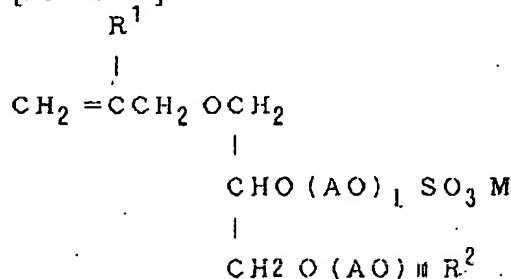
[0023] R1: H, CH₃, R2: Amino groups, such as unsubstituted or a permutation hydrocarbon group, the alkylene group of A:C 2-4, n:0-100M:univalent, a divalent cation, [0024]

[Formula 6]



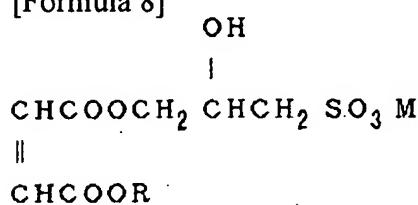
[0025] R1: R3:H and propenyl radicals, such as R2:H, alkyl groups of C6-18, etc., such as an alkyl group of C 6-18, the alkylene group of A:C 2-4, a permutation alkylene group, M: n:1-200, such as alkali metal, [0026]

[Formula 7]



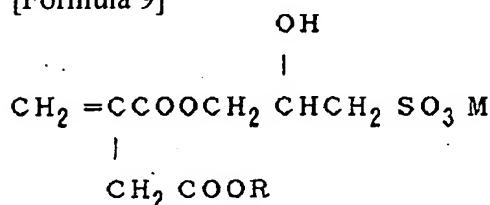
[0027] R1: L:0-20, such as alkylene groups of A:C 2-4, such as a hydrocarbon group of H, CH₃, and R2:C 8-24, M:H, alkali metal, and alkaline earth metal, m:0-50, [0028]

[Formula 8]



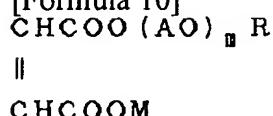
[0029] R: Eight to C32 hydrocarbon group, M : alkali metal, ammonium, [0030]

[Formula 9]



[0031] R: Eight to C22 hydrocarbon group, M : alkali metal, ammonium, [0032]

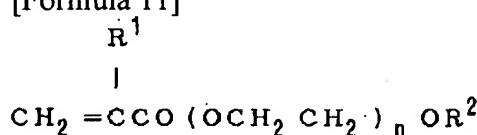
[Formula 10]



[0033] R: Alkyl, alkylphenyl, A:ethylene, M : ammonium, an amine, alkali metal, m:9, 12, 14, 28

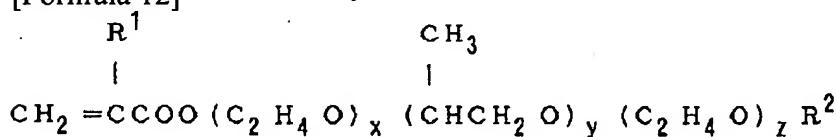
[0034]

[Formula 11]



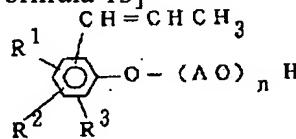
[0035] R1: H, CH₃, R2:H, CH₃, -C₆H₄-(CH₂)_m-H, n:4-30[0036]

[Formula 12]



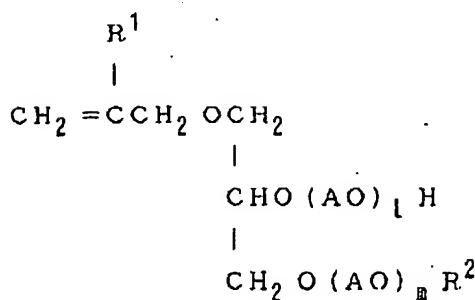
[0037] R1, R2:H, CH₃, x:0-100, y:0-10z:0-100, 1 <= x+y+z <= 100, [0038]

[Formula 13]



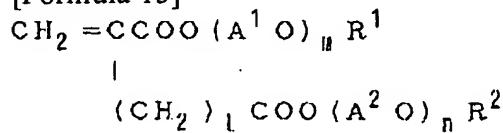
[0039] R1: R3:H and propenyl radicals, such as R2:H, alkyl groups of C6-18, etc., such as an alkyl group of C 6-18, the alkylene group of A:C 2-4, a permutation alkylene group, n:1-200, [0040]

[Formula 14]



[0041] R1: The hydrocarbon group of H, CH3, and R2:C 8-24, an acyl group, the alkylene group of A:C 2-4, L:0-100, m:0-50, [0042]

[Formula 15]



[0043] R1, R2:H, the hydrocarbon group of C1-36, an acyl group, A1, the alkylene group of A2:C 2-4, a permutation alkylene group, and L: -- when each of 1, 2m, n:0, positive numbers, m+n>=3, and R1 and R2 is H, reactant nonionic surfactants, such as m and n>=1, etc. are mentioned.

[0044] In addition, a nonresponsive surfactant can also be used together in order to improve the stability of an emulsion. However, since a water resisting property is reduced, the amount is used, restricting. As a nonresponsive emulsifier, nonionic surface active agents, such as cationic surface active agents, such as anionic surface active agents, such as alkyl or an alkyl allyl compound sulfate, alkyl or an alkyl allyl compound sulfonate, and dialkyl sulfo succinate, alkyl trimethylammonium chloride, and alkyl benzyl ammoniumchloride, polyoxyethylene alkyl phenyl ether, polyoxyethylene alkyl ether, and polyoxyethylene carboxylate, etc. are illustrated.

[0045] As a radical initiator in a polymerization, if used for the usual emulsion polymerization, all can be used and oil solubility types, such as water-soluble types, such as a hydrochloride of persulfate systems, such as potassium persulfate and ammonium persulfate, a hydrogen peroxide, t-butyl hydro peroxide, and azobis amidinopropane, benzoyl peroxide, cumene hydro peroxide, dibutyl peroxide, diisopropyl peroxide, cumil peroxy neodecanoate, cumyl peroxy octoate, and azobisiso BUCHINI tolyl, etc. will be mentioned. The redox system which furthermore combined reducing agents, such as sodium hydrogen sulfite, a Rongalite, and an ascorbic acid, with the initiator can also be used.

[0046] The usual emulsion polymerization is sufficient as a polymerization, and the approach which emulsifies a monomer, water, and an emulsifier beforehand in the batch type which bundles up all monomers and is taught to a reaction can, the dropping type which trickles a monomer at any time during a reaction, and a dropping type, and is dropped as a monomer emulsion can be used for it. By request, additives, such as the chain transfer agent used for adjustment of the polymerization degree for the improvement in gloss, the film formation assistant which adjusts minimum film forming temperature, a plasticizer which improves the flexibility of a paint film, a thickener, a defoaming agent, antiseptics, and an antifreezing agent, can be used.

[0047]

[Embodiment of the Invention] The emulsion of this invention carries out emulsification distribution of a monomer and the water using a surface active agent beforehand, creates a monomer emulsion, trickles the monomer emulsion created previously into the reaction can which subsequently prepared and carried out temperature up of water and the surface active agent, it adds an initiator suitably, carries out an emulsion polymerization, and is obtained.

[0048]

[Example]

With an example 1 reaction can, the water 445 weight section, the sodium acetate 2.5 weight section, the anionic polymerization nature surfactant 1 weight section, The nonionic polymerization nature surfactant 8 weight section and the anionic surfactant 2.5 weight section are taught. To this, the water 484 weight section, the anionic polymerization nature surfactant 14 weight section, the nonionic polymerization nature surfactant 9.5 weight section, The anionic surface-active-agent 8.5 weight section, the styrene 423 weight section, the 2-ethylhexyl acrylate 417 weight section, What carried out emulsification mixing of the methylmetaacrylate 217 weight section, the 80% acrylic-acid water-solution 24.5 weight section, and the meta-acrylamide 28.5 weight section is dropped, temperature is controlled at 80 degrees C, the ammonium persulfate 61.5 weight section is dropped at an initiator 5%, and an emulsion polymerization is carried out to it. The aqueous ammonia after polymerization termination neutralized and the emulsion of 50% of nonvolatile matters was obtained. Tg on count of the obtained emulsion was 11.5 degrees C.

[0049] Based on the polymerization presentation of two to example 9 table 1, others obtained each emulsion like the example 1.

[0050]

[A table 1]

		実験例								
		1	2	3	4	5	6	7	8	9
仕込み	水	445	445	445	477	455	463	474	445	445
	酢酸ソーダ	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
	アニオン性重合性界面活性剤	1	1	1	1	1	1	1	1	1
	ノニオン性重合性界面活性剤 (8.0%水溶液)	8	8	8	8	8	8	8	8	8
	アニオン性界面活性剤 (5.0%水溶液)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
乳化モ	ノニオン性界面活性剤 (8.0%水溶液)									
	水	484	484	484	484	484	484	484	484	484
	アニオン性重合性界面活性剤	14	14	14	14	14	5	5	5	5
	ノニオン性重合性界面活性剤 (8.0%水溶液)	9.5	9.5	9.5	9.5	9.5	9.5	9.5	9.5	9.5
	アニオン性界面活性剤 (5.0%水溶液)	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5
ノ	ノニオン性界面活性剤 (8.0%水溶液)									
	ステレン	423	423	423	423	423	423	423	423	423
	2-エチルヘキシル	417	417	417	417	417	417	417	454	370
	アクリレート									
	メチルメタアクリレート	217	217	217	217	217	217	217	180	264
1	8.0%アクリル酸	24.5	24.5	24.5	24.5	24.5	24.5	24.5	24.5	24.5
	メタクリルアミド	28.5	15.9	52.9	28.5	28.5	28.5	28.5	28.5	28.5
	アセトアセトキシ				31.7		31.7	31.7		
	エチルメタクリレート								10.6	
	グリジルメタクリレート									10.6
合計		2085	2072.4	2119.4	2148.7	2105.6	2125.7	2147.3	2076	2076

[0051] The example 1 (example which did not use an amide group content acrylic monomer) of a comparison

Based on the polymerization presentation of a table 2, others obtained each emulsion like the example 1.

[0052] The example 2 (example which did not use a polymerization emulsifier) of a comparison

Based on the polymerization presentation of a table 2, others obtained each emulsion like the example 1.

[0053] The example 3 (example which used the amide group content acrylic monomer too little) of a comparison

Based on the polymerization presentation of a table 2, others obtained each emulsion like the example 1.

[0054] The example 4 (example which used the amide group content acrylic monomer superfluously) of a comparison

Based on the polymerization presentation of a table 2, others obtained each emulsion like the example 1.

[0055]
[A table 2]

		比 較 例			
		1	2	3	4
仕 込 み	水	445	432	445	445
	酢酸ソーダ	2.5	2.5	2.5	2.5
	アニオン性重合性界面活性剤	1		1	1
	ノニオン性重合性界面活性剤 (8.0%水溶液)	8		8	8
	アニオン性界面活性剤 (5.0%水溶液)	2.5	4.6	2.5	2.5
	ノニオン性界面活性剤 (8.0%水溶液)		9.9		
乳 化 モ ド	水	484	484	484	484
	アニオン性重合性界面活性剤	14	11	14	14
	ノニオン性重合性界面活性剤 (8.0%水溶液)	9.5		9.5	9.5
	アニオン性界面活性剤 (5.0%水溶液)	8.5	14.7	8.5	8.5
	ノニオン性界面活性剤 (8.0%水溶液)		12.1		
	ステレン	423	423	423	423
ノ マ 1	2-エチルヘキシル アクリレート	417	417	417	417
	メチルメタアクリレート	217	217	217	217
	8.0%アクリル酸	24.5	24.5	24.5	24.5
	メタクリルアミド		28.5	3.2	116
	アセトアセトキシ エチルメタクリレート				
	グリシジルメタクリレート				
合 計		2056.5	2074.6	2059.7	2172.5

[0056] The monolayer elastic coating was created by the assessment (creation of coating) following coating formula.

Water 74 weight sections Ethylene glycol 14 weight sections Pigment agent The 9.2 weight sections
Antiseptics Two weight sections Defoaming agent 1 weight section Titanium oxide 93 weight sections
Calcium carbonate 93 weight sections -- grinding of this is carried out by the sand mill, and it is
considered as a pigment paste. To a degree Emulsion The 565 weight sections 14% aqueous ammonia
The 5.5 weight sections Butyl cellosolve/water (1/2) 84 weight sections TEKISA Norian 44 weight
sections Defoaming agent Two weight sections Acrylic-acid system thickener water solution 28 weight
sections Urethane system thickener water solution 24 weight sections were added and agitated.

(Assessment trial)

(1) Carry out coating of the coating to the slate plate which carried out coating of the initial water
resisting property solvent system chlorinated-polyolefins sealer of desiccation by 2mm thickness (wet),
dry at a room temperature for 24 hours, and check whether water immersion is carried out for 24 hours,
and there is neither runoff of a paint film nor generating of a blister.

The spill of a paint film, those of a blister with generating With the spill of x paint film, and no
generating of a blister O (2) With a roller, the slate plate which carried out coating of the acrylic
sealer of a raindrops-proof stain resistance emulsion mold is painted twice, it is made it, and coating is
carried out to the pattern pattern of an average of 2mm thickness (wet). It is made to dry for one week at
a room temperature, and installs in an outdoor raindrops testing device. (Raindrops testing device: In a
slate plate, about 10 degrees is a mounting-on slant beam thing from a right angle, and, as for in the case
of a rainfall, raindrops generate the corrugated plate made from plastics from the trough of a corrugated
plate in the upper part of a slate plate at a slate plate so that it may be easy to generate raindrops.)
clear -- those with raindrops contamination x -- almost with no raindrops contamination O
assessment result is shown in a table 3.

[0057]

[A table 3]

		実施例									比較例			
		1	2	3	4	5	6	7	8	9	1	2	3	4
評価	エマルジョン不揮発分 (%)	50	50	50	50	50	50	50	50	50	50	50	50	50
	Tg	11.5	11.3	13.5	12.2	11.5	12.2	12.6	5.2	19.3	9.3	11.5	9.6	17.7
価値	乾燥初期耐水性	○	○	○	○	○	○	○	○	○	○	×	○	×
	耐雨だれ汚染性	○	○	○	○	○	○	○	○	○	○	×	○	×

[0058]

[Effect of the Invention] The emulsion of this invention has very low raindrops stain resistance, and does so the outstanding effectiveness which has the high initial water resisting property which neither blistering of a paint film nor runoff produces by the rainfall after construction.

[Translation done.]